

PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
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PERIOD COVERED: May, 1990

I. COMMERCIALIZATION OF GMC AND POLYMIC

- A. Objective:** To find chemical manufacturers who are capable of producing large quantities of GMC and polyMIC.
- B. Results:** The production of batch 004 of GMC was carried out at Lee Labs. Approximately 215 pounds of product was produced (expected yield - 290 pounds). The lower yield was most probably due to losses associated with carbon treatment. In an attempt to obtain the same low color as in batch 003, the product was treated twice with carbon. The total amount of carbon used for this batch (two treatments) was of the order of 40 kilograms. With batch 003, a different filter was used and the amount of carbon was closer to 10 kilograms. In the future this excessive carbon treatment will be eliminated as it has been found that the yellow color obtained with GMC is not readily removed with carbon treatment. Analytical data is currently being determined on this material.

After the production of batch 004 we used the reactor to dilute approximately 30 gallons of 77% GMC (batch 003) to 61.5% GMC with absolute ethanol. Dilution went well with only minor heating needed to dissolve the GMC. It was noted that on storage for about 6 months, the color of the solution had an increased yellow cast.

An outline of the current status of the PolyMIC project was received from Callery Chemical. Work with potassium enolates is showing some success. Work with quaternary ammonium enolates has not been successful.

II. FLAVOR RELEASE TECHNOLOGY

- A. Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

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- B. **Results:** The large scale production of CR-2910 at Aldrich has been completed. A total of 190 lbs have been produced. Forty pounds were delivered to PM. The purity of the material is 99% by HPLC.

A research contract has been given to R.S.A. to develop a method for the synthesis of Vanillin Chloroformate. Method is to be suitable for large scale production of this material.

Methyl 4,6-O-(4'-t-butoxycarbonyloxy-3'-methoxybenzylidene)- α -D-glucopyranoside (t-BOC-Aromatek-215) was prepared from methyl 4,6-O-(4'-hydroxy-3'-methoxybenzylidene)- α -D-glucopyranoside (A-215) and di-t-butyl dicarbonate in high yield. We are in the process of attempting to crystallize this material. A sample of the crude material was delivered to Dr. Ted Sanders for stability evaluation.

Also prepared was divanillyl carbonate from vanillin and triphosgene. The material is only slightly soluble in ethanol at room temperature, but unlike the divanillyl oxalate, this material can be recrystallized from a variety of solvents. Preliminary pyrolysis GC/MS showed that at 300°C, the material pyrolyzed to a large extent to give carbon dioxide and vanillin.

III. FLAVOR CHEMISTRY

- A. **Objective:** To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.
- B. **Results:** We continue our work on the recovery of organic components from the ART process water column effluent. Different material, such as reversed-phase silica gel (C-18), are being tested.

About 60 liters of effluent were used to isolate organics for subsequent fractionation and identification. A sample of 35g material has been isolated and was fractionated on silica-gel to give eight fractions. Odor profile and subjective smoking will be used to identify the best fraction.

Work continues on the removal and recovery of nicotine from XAD-4 resin (polystyrene).

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